

We claim:

1. A process for the recovery of a Lewis acid from a reaction mixture (I) which has been obtained in the hydrocyanation of an olefinically unsaturated compound to a nitrile which has a miscibility gap with water under certain amount, pressure and temperature conditions, in the presence of a catalyst system comprising a Lewis acid and a complex compound comprising a phosphorus-containing compound which is suitable as ligand and a central atom which is suitable for this compound,
which comprises
 - a) removing the said complex compound from mixture (I) to give a mixture (II),
 - b) adding water to mixture (II) and placing the latter under pressure and temperature conditions such that a phase (III) which has a higher content of water than of the said nitrile and a phase (IV) which has a higher content of the said nitrile than of water are obtained, where phase (III) has a higher content of the said Lewis acid than does phase (IV),
 - c) adding a liquid diluent (V) which
 - c1) does not form an azeotrope with water and whose boiling point under certain pressure conditions is higher than that of water or
 - c2) forms an azeotrope or heteroazeotrope with water under certain pressure conditions,
 - d) to phase (III),
 - e) subjecting the mixture of phase (III) and liquid diluent (V) to distillation under the pressure conditions mentioned in step c1) or c2), giving a mixture (VI) which has a higher content of water than of diluent (V) and a mixture (VII) which has a higher content of diluent (V) than of water, where mixture (VII) has a higher content of the said Lewis acid than does mixture (VI),
and

- 5 e) subjecting mixture (VII) to hydrocyanation of an olefinically unsaturated compound to give a nitrile which has a miscibility gap with water under certain amount, pressure and temperature conditions, in the presence of a catalyst system comprising a Lewis acid and a complex compound comprising a phosphorus-containing compound which is suitable as ligand and a central atom which is suitable for this compound.
- 10 2. A process as claimed in claim 1, where mixture (VII) has a water content of less than 0.5% by weight, based on mixture (VII).
- 15 3. A process as claimed in claim 1 or 2, where the solubility of the said Lewis acid in diluent (V) under the distillation conditions in step d) is at least 0.1% by weight, based on diluent (V).
- 20 4. A process as claimed in any one of claims 1 to 3, where step b) is carried out in countercurrent in a multistage extraction column.
- 25 5. A process as claimed in any one of claims 1 to 4, where all or some of mixture (VI) is fed back into step b).
- 30 6. A process as claimed in any one of claims 1 to 5, where the water employed in step b) has a pH of less than 7.
- 35 7. A process as claimed in any one of claims 1 to 5, where the water employed in step b) has a pH in the range from 0 to less than 7.
- 40 8. A process as claimed in any one of claims 1 to 7, where an acid is added to the water employed in step b).
- 45 9. A process as claimed in claim 8, where HCl is added to the water.
10. A process as claimed in any one of claims 1 to 9, where the diluent (V) contains all or some of the compound to be hydrocyanated in step e).
11. A process as claimed in any one of claims 1 to 10, where the diluent (V) employed is a nitrile selected from the group consisting of 2-cis-pentenitrile, 2-trans-pentenitrile, 3-cis-pentenitrile, 3-trans-pentenitrile,

18

4-pentenitrile, E-2-methyl-2-butenitrile, Z-2-methyl-2-butenitrile, 2-methyl-3-butenitrile or a mixture thereof.

12. A process as claimed in any one of claims 1 to 11, where all
5 or some of the undissolved constituents are separated off
from mixture (II) between steps a) and b) or between steps b)
and c).

10

15

20

25

30

35

40

45